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**National Environmental Testing**

**Dayton Division**

US EPA RECORDS CENTER REGION 5



471568

**Standard Operating Procedure**

Analyte or Suite: Total Suspended Solids (TSS)  
Total Volatile Suspended Solids (TVSS)

Methodology: Gravimetric

Reference: EPA 600/4-79-020 Method 160.2  
Standard Methods, 18th Edition, Method 2540D  
Standard Methods, 18th Edition, Method 2540E

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## **1. Introduction and Scope**

### **1.1. General**

Preservation: 4 degrees C

Container: 1 liter glass or plastic

Holding Time: 7 days

Range of Test: 3 mg/L - 20,000 mg/L

Nominal Reporting Limit: 3 mg/L

1.2. Solids refer to matter suspended or dissolved in water or wastewater. Solids may affect water or effluent quality adversely in a number of ways. Waters high in suspended solids may be esthetically unsatisfactory for such purposes as bathing. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations.

1.3. "Total suspended solids" (TSS) is the term applied to the residue retained by a glass fiber filter and dried to a constant weight at 103-105 degrees C. Total suspended solids is also known as nonfilterable residue.

1.4. This method is applicable to drinking, surface, and saline water, domestic and industrial wastes.

1.5. "Fixed solids" is the term applied to the residue of total, suspended, or dissolved solids after heating to dryness for a specified time at a specified temperature. The weight loss on ignition is called "volatile solids." Determinations of fixed or volatile solids do not distinguish precisely between inorganic and organic matter. It includes losses due to decomposition or volatilization of some mineral salts.

## **2. Summary of Method**

A well-mixed sample is filtered through a weighed standard glass fiber filter and the residue retained on the filter is dried to a constant weight at 103 to 105 degrees C. The increase in weight of the filter represents the total suspended solids. The difference between the total solids and the total dissolved solids is defined as the total suspended solids.

### 3. Safety

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards, and all associated protective wear and spill clean-up procedures PRIOR TO THE USE of any chemical. In all cases, both the applicable MSDS and supervisor or Safety Officer should be consulted. The employee should comply with all safety policies as presented in the NET Safety Manual. The bottle labels also provide important information that must be noted. If you have any questions, consult your supervisor or safety officer.

Personnel performing this procedure may be working with flammables, poisons, toxics, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and labcoats must be worn, and solvents will be handled in ventilated hoods, in addition to other measures prescribed by the Division. It should be noted that samples must be handled with as much care as any of the materials used in this method due to the unknown nature of their composition.

### 4. Reagents and Materials

#### 4.1. Apparatus.

The following apparatus is recommended for performing this procedure. Equivalent items should only be used as a last resort or when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met.

4.1.1. Glass Fiber filters: Whatman 934-AH, 4.25 cm.

4.1.2. Aluminum pans, weighing or equivalent.

4.1.3. Muffle furnace for operation at  $550 \pm 50$  degrees C.

4.1.4. Desiccator, provided with a desiccant containing a color indicator of moisture concentration. A mixture of Drierite Indicating Desiccant and Drierite Non-indicating Desiccant should be used.

4.1.5. Drying oven, for operation at 103 to 105 degrees C.

4.1.6. Analytical balance, capable of weighing to 0.1 mg.

4.1.8. Filtration apparatus: manifold system able to accept magnetic polysulfone filter funnels or equivalent.

4.1.9. Graduated cylinders, various sizes.

#### **4.2. Reagents.**

The following reagents are required to perform this procedure. When instructions are given on how to prepare a specific volume of a reagent, larger or smaller volumes can be prepared as needed so long as the final concentrations remain the same. Any other deviation from the reagents used in this SOP could be detrimental to the quality of the data produced.

All reagents must be properly labeled with the reagent identification and concentration, date prepared, expiration date, initials of analyst, and applicable safety information. The label has a place for the NFPA diamond, which will be used to indicate health (blue), flammability (red), reactivity (yellow), and contact/special (white) information obtained from applicable Material Safety Data Sheets (MSDS) supplied by the vendor.

4.2.1. Deionized water: Prepare by passing water through a mixed bed of cation and anion exchange resins or an equivalent source. Use deionized water for the preparation of all reagents, calibration standards, and dilution water.

#### **4.3. Standards.**

The following standards are recommended for performing this procedure. The use of alternative standards will be allowed as long as they are of equal or greater quality and there is an associated improvement in efficiency, productivity, or cost. When instructions are given on how to prepare a specific volume of standard, larger or smaller volumes can be prepared as needed so long as the volumes used are properly documented.

4.3.1. Class S weights shall be used to check the analytical balance daily.

4.3.2 TSS Standard: Celite Ashless Powder (C212-500). 100 mg/L TSS = 10 mg diluted to 100 mL. Analyze 100 mL. Prepare monthly. Shake vigorously before use, the powder tends to settle very quickly.

### **5. Interferences**

5.1. Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not desired in the final result. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg residue. For samples high in dissolved solids thoroughly wash the filter to ensure removal of the dissolved material. Prolonged filtration times resulting from filter clogging may produce high results owing to excessive solids capture on the clogged filter.

## **6. Analytical Procedures**

### **6.1. Preservation and Handling.**

Samples may be collected in plastic or glass, and must be stored at 4 degrees C until time of analysis. The maximum holding time for TSS is 7 days.

### **6.2. Instrument Calibration.**

Calibrate the analytical balance according to the manufacturer's specifications.

### **6.3. Sample Analysis.**

6.3.1. Preparation of glass-fiber disk. Insert disk with wrinkled side up in filtration apparatus. Apply vacuum and wash disk with three successive 20 mL portions of distilled water. Continue suction to remove all traces of water, and discard washings. Remove filter from filtration apparatus and transfer to an aluminum dish and place in a drying oven at 103-105 degrees C until completely dry (one hour minimum drying time). Store prepared filter and aluminum pan in oven until needed. Prior to use, place in desiccator and allow to cool. Weigh immediately before use.

6.3.2. Selection of sample sizes. Choose sample volume to yield between 2.5 and 200 mg dried residue. If more than 10 minutes are required to complete filtration, discard sample aliquot and filter and stop the analysis. Begin analysis again using a smaller sample volume.

6.3.3. Pre-weigh an aluminum pan with filter. Place filter in the filtering apparatus and begin suction. Wet filter with a small volume of distilled water to seat it. Filter a measured volume of well-mixed sample through the glass fiber filter. Rinse the graduated cylinder with three successive 10-mL volumes of distilled water. Similarly, wash the inside of the filtration apparatus with three successive 10 mL washings, allowing complete drainage between washings and continue suction for about 1 min after filtration is complete. Carefully remove filter from filtration apparatus and transfer to the corresponding pre-weighed aluminum dish. Dry for at least 2-4 hr at 103 to 105 degrees C in an oven, cool in a desiccator to balance temperature, and weigh.

Note: If sufficient vacuum is not applied to the filter and it remains "wet", the filter will stick to the bottom of the pan upon drying.

#### 6.4. Calculation.

$$\text{TSS (mg/L)} = \frac{(A - B) \cdot 1000}{V}$$

where:

A = weight of filter + dried residue, mg

B = weight of filter, mg

V = sample volume, mL

#### 6.5. Volatile Suspended Solids. (If Required)

6.5.1. Pre-heat muffle furnace to  $550 \pm 50$  degrees C.

6.5.2. Remove filter from aluminum pan and place in a porcelain crucible. Save aluminum pan.

6.5.3. Ignite residue in muffle furnace 20 minutes.

6.5.4. Allow crucible to cool. Remove filter from crucible and return to original aluminum pan. Reweigh aluminum pan and filter.

#### 6.6. Calculation.

$$\text{mg/L VSS} = \frac{(A-B) \times 1000}{\text{Sample Volume, mL}}$$

where:

A = weight of filter and pan before ignition, mg

B = weight of filter and pan after ignition, mg

#### 6.7. Constant Weight

Filters must be dried to constant weight. Achievement of constant weight can be demonstrated by repeated cycles of drying, cooling, desiccating, and weighing until constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less. Drying the samples for 2-4 hours will normally ensure attainment of constant weight. Actual documentation of this fact should be on file for reference for a representative set of samples. If less time is allowed for drying the filter from a batch, resort to actually performing the constant weight determinations for all samples in the batch.

### **7. Quality Control.**

The following details the QC requirements which apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either instrument performance, method performance (including sample preparation), or individual sample performance. Our goal is to produce data of unquestionable quality. Always remember what purpose the QCI serves when evaluating QCI results. Guidelines can be provided, and are

provided, but they cannot take the place of a logistical, common-sense evaluation of the complete data set.

#### 7.1. Method Detection Limits and Reporting Limits.

An MDL study, following the MDL SOP, must be done during initial method validation and then annually. If the analytical method is changed, an MDL study must be done again. The calculated MDL must not exceed the reporting limit. The current nominal reporting limit for this parameter is 10 mg/L.

#### 7.2. Method Validation Sample (MVS).

##### 7.2.1. Definition and Use of MVS

The purpose of the MVS is to verify and demonstrate that the method and/or analyst is capable of generating precise and accurate analytical data. Method validation samples consist of four replicate aliquots of spiked deionized water prepared and analyzed in a manner identical to samples. The spike concentration should be at the mid range of the analysis. The samples should be prepared and analyzed in the same batch. They are used to validate new analyst and new instrument performance, and to validate changes in analytical equipment or techniques. MVS requirements are built into NET's internal analyst certification protocols.

##### 7.2.2. Frequency of MVS

Method validation must be repeated whenever a significant change in the method or instrumentation is made which could cause the previous MVS to become invalidated. Also, they will be routinely analyzed as part of training and certification of analysts newly performing the analysis.

##### 7.2.3. Criteria for MVS

The average percent recovery should pass the interim acceptance criteria applied to the LCS, which is 80-120%, and the relative standard deviation should be within  $\pm 20\%$ .

##### 7.2.4. Corrective Action for MVS

If a problem is indicated, it must be identified and corrected, and if necessary, MVSS must be re-prepared and re-analyzed. If the problem involves only the instrument, the MVSS must be re-analyzed.

##### 7.2.5. Documentation

Since the MVSS serve several purposes, results of the method validation should be filed either with individual analyst training records, method validation records, or with instrument validation records. How they are filed is dependent on the reason for the study being performed. An alternative would be to

file the results jointly and cross reference other files as appropriate. In either case, the data must be retrievable.

### 7.3. Analyst Certification.

Each analyst performing this method must successfully complete the requirements detailed in the certification SOP. A performance sample, which is administered by the QA coordinator or the Department Supervisor must to be successfully analyzed.

### 7.4. Calibration Curve.

Not Applicable

### 7.5. Initial Calibration Verification Standard (ICVS).

#### 7.5.1. Definition and Use of ICVS

The purpose of the ICVS is to verify that the balance has been properly calibrated. Ultimately, Class S weights are normally utilized for this purpose.

#### 7.5.2. Frequency of ICVS

On a daily basis the balance calibration is verified with the use of two class "S" weights, one in the milligram range and one in the gram range.

On a monthly basis the balance is calibrated against a class "S" set of weights over the range of the balance capability.

#### 7.5.3. Criteria for ICVS

(Known) indicates a statistical range of 3 standard deviations of 30 or more readings for the "known" weight. This weight does not need to be class "S".

#### 7.5.4. Corrective Action for ICVS

If the (known) weight does not meet the acceptable range then the weight should be cleaned and re-weighed. If the reading is still outside the established control criteria, then the QC coordinator should be contacted to re-calibrate and check the balance against the class "S" set of weights.

#### 7.5.5. Documentation

Record the daily (known) weight reading in the Balance Calibration Record and/or on the raw data.

### 7.6. Reagent Blank (RB).

Since TSS samples, blanks, and standards must be processed through the filtration, the reagent blank (RB) and the procedure blank (PB) are equivalent.

See Section 7.8.

7.7. Continuing Calibration Verification Standard (CCVS).

Since TSS samples, blanks, and standards must be processed through the filtration, the CCVS and LCS are equivalent.

See Section 7.9.

7.8. Preparation Blank (PB).

7.8.1. Definition and Use of PB

The preparation blank is a deionized water blank that is subjected to the same conditions that a sample undergoes. The preparation blank is used to demonstrate method performance. A "clean" preparation blank demonstrates that the preparation procedure is free of contamination.

Note: Since TSS samples, blanks and standards must be processed through the filtration, the reagent blank (RB) and the procedure blank (PB) are equivalent.

7.8.2. Frequency of PB

Analyze a minimum of one procedure blank per preparation batch. A batch shall contain twenty samples or less.

7.8.3. Criteria for PB

Acceptance criteria requires the procedure blank to be less than the reporting limit.

Procedure blanks are not routinely subtracted from the analytical results.

7.8.4. Corrective Action for PB

If a preparation blank shows a detection above the reporting limit for a parameter, then the sample batch must be re-prepared.

If positive values below the reporting limit are observed, they should be evaluated in relation to the sample(s) and extra care should be taken to avoid reporting false positives.

7.8.5. Documentation

Record the concentration of the preparation blank on the raw data or in the lab book.

Enter the preparation blank result into LABSYS2 in the blank entry.

## 7.9. Lab Control Standard (LCS).

### 7.9.1. Definition and Use of the LCS

The lab control standard is a standard that is subjected to the same conditions that a sample undergoes. The LCS analysis is designed to serve as a monitor of the efficiency of the entire procedure.

Note: Since TSS samples, blanks, and standards must be processed through the filtration, the C CVS and LCS are equivalent.

### 7.9.2. Frequency of LCS

Analyze a minimum of one LCS per preparation batch. A batch shall be twenty samples or less.

### 7.9.3. Criteria of LCS

Interim acceptance criteria requires the LCS to be within 80-120% of the true value.

After a data base of 20-30 points has been collected, calculate the mean expressed as percent recovery and the standard deviation (s).

$$\begin{aligned}\text{Upper Control Limit (UCL)} &= \text{mean} + 3s \\ \text{Upper Warning Limit (UWL)} &= \text{mean} + 2s \\ \text{Lower Warning Limit (LWL)} &= \text{mean} - 2s \\ \text{Lower Control Limit (LCL)} &= \text{mean} - 3s\end{aligned}$$

The control limits and warning limits are updated yearly or whenever the process is changed. The data must be plotted on a control chart. The purpose of control charting is to obtain real-time trend analysis of method performance.

### 7.9.4. Corrective Action for LCS

The inability of the laboratory to successfully analyze the LCS indicates a problem potentially related to the sample preparation procedures. If the control windows are exceeded, all sample results associated with the LCS are suspect and should be re-prepared and reanalyzed, after the cause of the problem has been determined and corrected. If reanalysis of the sample occurs outside holding times or if insufficient sample is available for reanalysis, the results must be flagged and the LCS reported to the client.

### 7.9.5. Documentation

Record the percent recovery on the raw data or in the lab book. Enter the percent recovery of the LCS into LABSYS2 in the LCS entry.

## 7.10. Duplicate.

### 7.10.1. Definition and Use of Duplicate

The duplicate is a separate aliquot of sample subjected to the same conditions that a sample undergoes. These data are generated to determine long-term precision of the analytical method on various matrices. These data alone cannot be used to evaluate the precision of individual samples, except for the sample chosen for the duplicate analysis.

### 7.10.2. Frequency of Duplicate

Analyze a minimum of one duplicate per every ten samples.

### 7.10.3. Criteria for Duplicate

Advisory interim acceptance criteria requires the duplicate relative percent difference to be less than 20.

After a data base of 20-30 points of a given matrix, i.e. aqueous and soils, have been collected, calculate the mean expressed as the absolute relative percent difference (RPD) the standard deviation (s).

$$\begin{aligned}\text{Upper Control Limit (UCL)} &= \text{mean} + 3s \\ \text{Upper Warning Limit (UWL)} &= \text{mean} + 2s\end{aligned}$$

The control limit and warning limit are updated yearly or whenever the process is changed. The data must either be tabulated or plotted on a control chart. These are advisory limits used for original and duplicate sample values that are greater than 5x the reporting limit of the parameter.

If the original and duplicate values are <5x the reporting limit, then the advisory control limit becomes  $\pm$  the reporting limits.

### 7.10.4. Corrective Action for Duplicate

No action is taken on out of control duplicate data alone to qualify an entire batch. Action taken must be weighed carefully since it may be difficult to determine if poor precision and/or accuracy is a result of sample non-homogeneity/uniqueness, method defects, or laboratory technique. However, the data may be used in conjunction with other QC criteria to determine the need for qualifying the data. If the duplicate data is outside acceptance limits, check percent recovery for the LCS. If the LCS is in control, the procedure is in control and the data is acceptable. Potentially, a matrix problem exists. Additional steps may be taken to determine the extent of the matrix interference. If the poor precision cannot be attributed to matrix, then the sample(s) should be reanalyzed.

#### 7.10.5. Documentation

The data generated can be presented, if necessary, as a statement of precision for a particular analysis on a given matrix. Record the RPD of the original and the duplicate value on the raw data or in the lab book. Enter the RPD result in LABSYS2 into the duplicate entry.

#### 7.10. Statistical Control Windows vs. Specifications

Within this section all of the QCIs listed have specified acceptance criteria, such as the LCS must be within  $\pm 20\%$ . In some cases, this specification is listed as "interim" and instructions are provided which require the generation of statistical control limits. If the generated statistical control limits are wider than the specification, the process should be questioned and carefully evaluated. If the process is not controlled, it is impossible to generate "tight" statistical windows. Occasionally, the process is in control but bias exists within the procedure.

### 8. References

8.1. Methods for Chemical Analysis of Water and Wastes, USEPA, Environmental Monitoring and Support Laboratory EPA-600/4-79-020

8.2. Standard Methods For the Examination of Water and Wastewater, 18th Edition, 1995

8.3. Federal Register 40 CFR Part 136.

8.4. Pre-existing internal documents (SOPs) from the various NET divisions were used as resources/references during the preparation of this document. These documents are on file at the corporate office.